



Creep/Stress Rupture Behavior and Failure Mechanisms of Full CVI and Full PIP SiC/SiC Composites at Elevated Temperatures in Air

R.T. Bhatt# and J. D. Kiser
NASA Glenn Research Center
Cleveland, OH
#Ohio Aerospace Institute

Support from NASA Fundamental Aeronautics Program

**Prepared for Presentation at the PACRIM 12 Conference, Waikoloa, Hawaii,
May, 2017**



Objective and CMC Materials

Objective:

- **Short term**
Determine creep behavior/creep rupture life/failure modes of full CVI and PIP SiC/SiC composites from 1315°C to 1450°C in air.
- **Long term**
Determine creep parameters of full CVI and PIP SiC/SiC composites to model and validate creep behavior of SOA SiC/SiC composites with (CVI+PIP) hybrid matrix.

Composite Materials:

Full CVI SiC/SiC(I): Rolls Royce HTC Inc, (Long Beach CA)

Full CVI SiC/SiC(II): GE Ceramic Composite Products (Newark, DE)

Full PIP SiC/SiC(I) & (II) : COIC Inc. (San Diego, CA)

Composite Characteristics:

Fiber/Weave: Sylramic-iBN SiC fiber, 5 Harness Satin Weave, 20 ends/inch.

Layup: 8 and 10 ply, 0°/90°

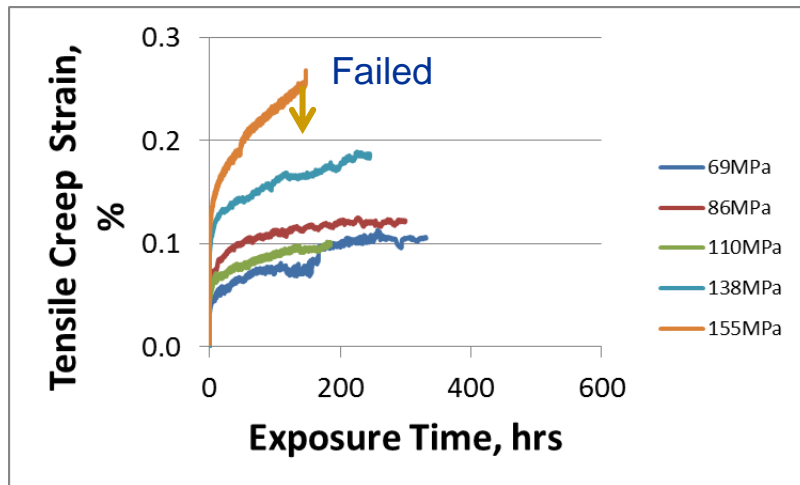
Interface: Chemical Vapor Infiltrated CVI BN and Synterial Mod1

Matrix: CVI SiC and PIP SiC

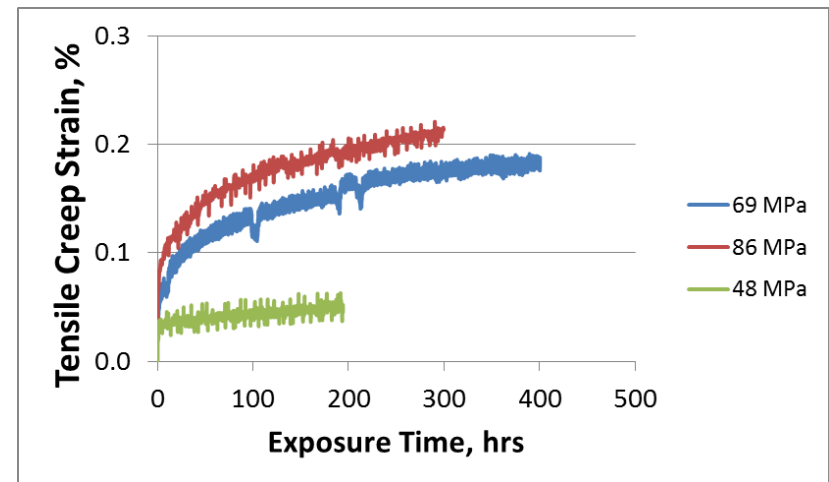
Note: Most test full CVI SiC/SiC specimens were CVI SiC seal coated and annealed at NASA.
The iBN treatment of Sylramic SiC cloth was conducted at NASA

Influence of Stress on Creep Behavior of Full CVI SiC/SiC Composites at 1315^oC and 1450^oC in air

CVI SiC/SiC(I) at 1315^oC



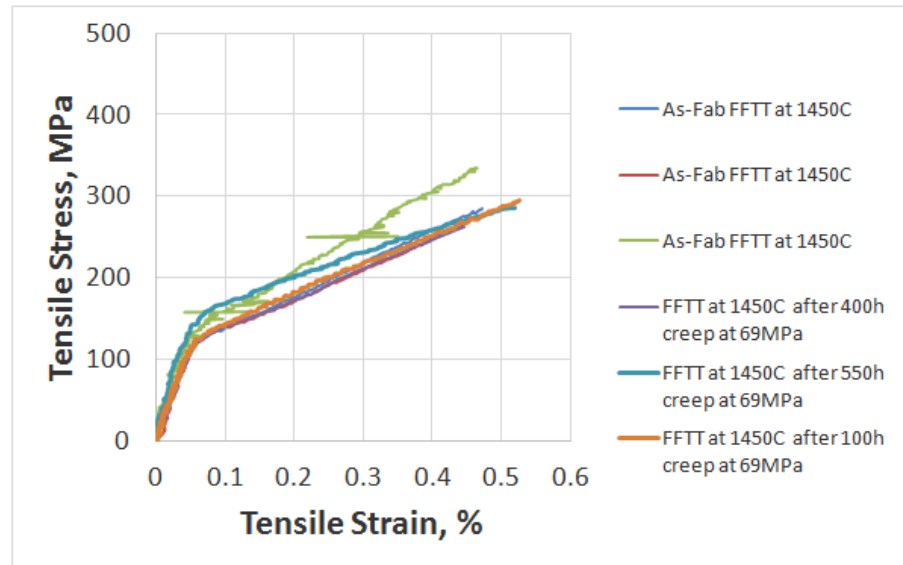
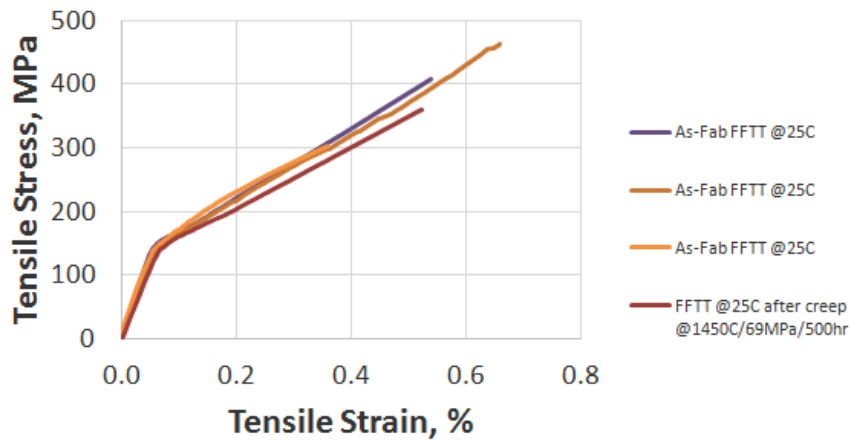
CVI SiC/SiC(II) at 1450^oC



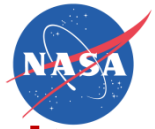
At low stresses creep strain vs. time plots show pseudo parabolic behavior. Only one specimen in the above plots failed during creep deformation. All specimens were creep tested to 250 to 400hrs without creep rupture.



Influence of Creep on Room Temperature and 1450°C Tensile Stress-Stain Behavior of Full CVI SiC/SiC Composites

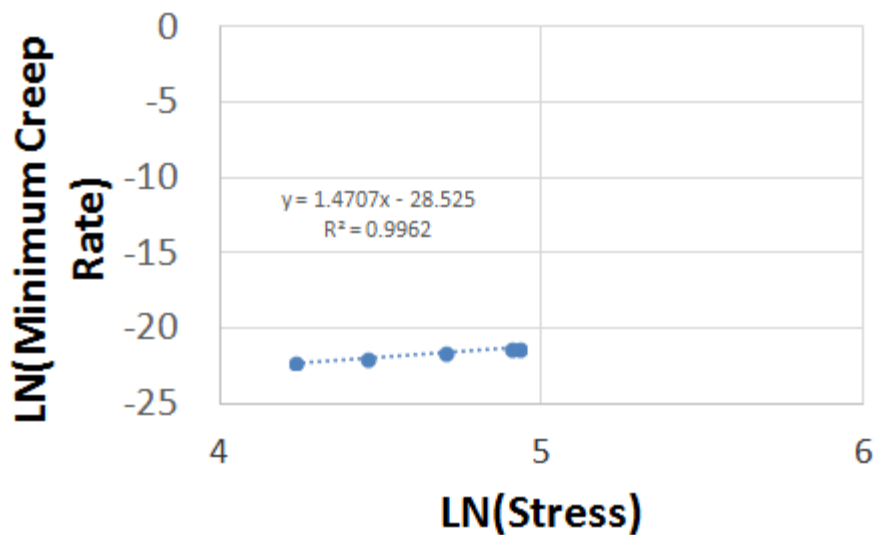


Crept specimens retain significant % of in-plane tensile properties of as-fabricated composites

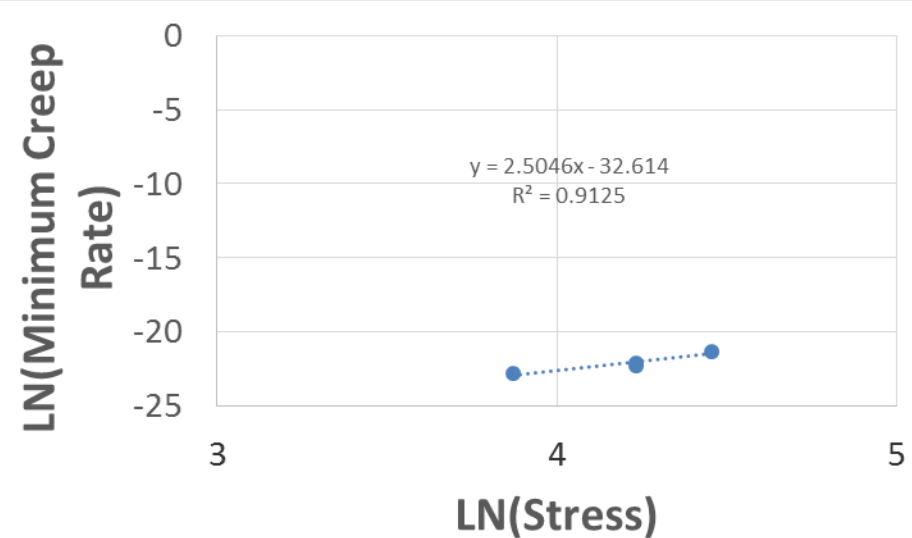


Stress Exponents of Creep for Full CVI SiC/SiC Composites at 1315°C and 1450°C

CVI SiC/SiC(I) at 1315°C



CVI SiC/SiC(II) at 1450°C



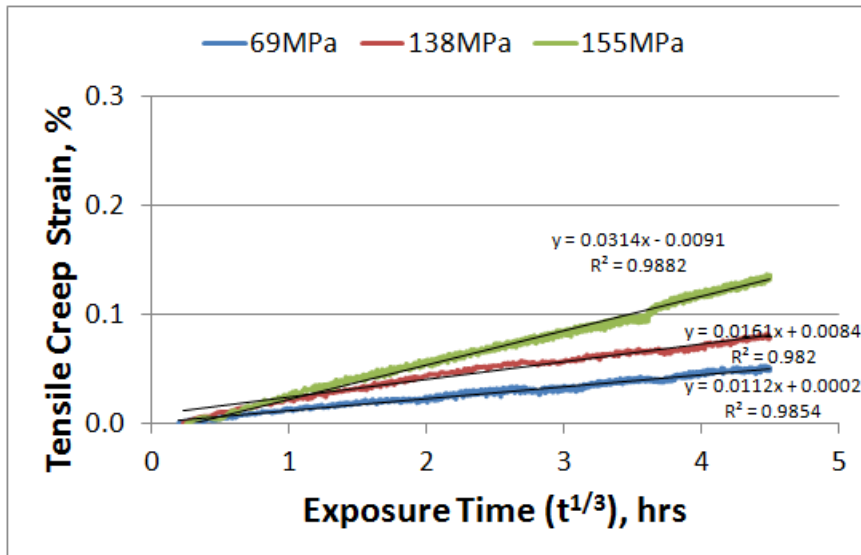
From 1315°C to 1450°C, the stress exponents of creep vary from ~1.5 to 2.5. This variation is possibly due to compositional variation of CVI SiC matrix fabricated by different vendors.

For measuring stress exponents, creep data of specimens that survived >250hr exposure were chosen.

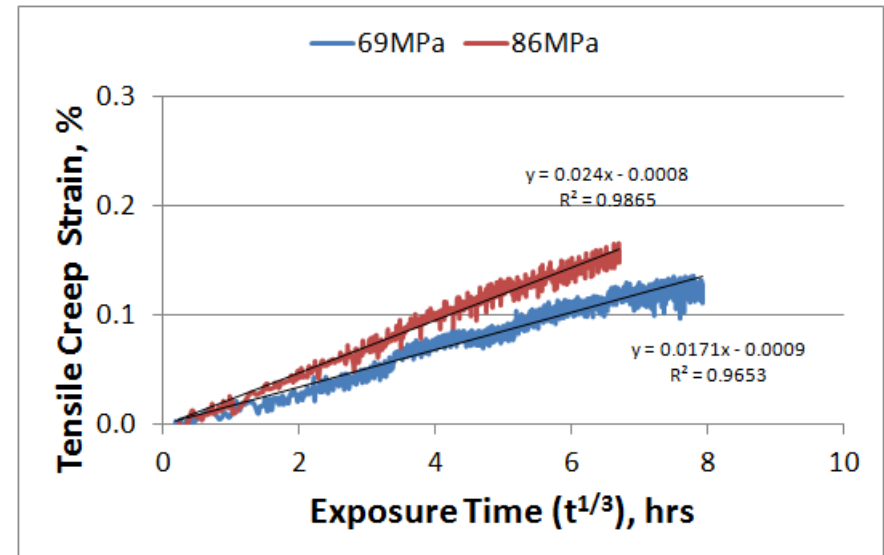


Time Dependency of Creep for Full CVI SiC/SiC Composites at 1315°C and 1450°C

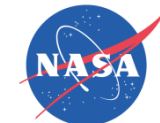
CVI SiC/SiC(I) at 1315°C



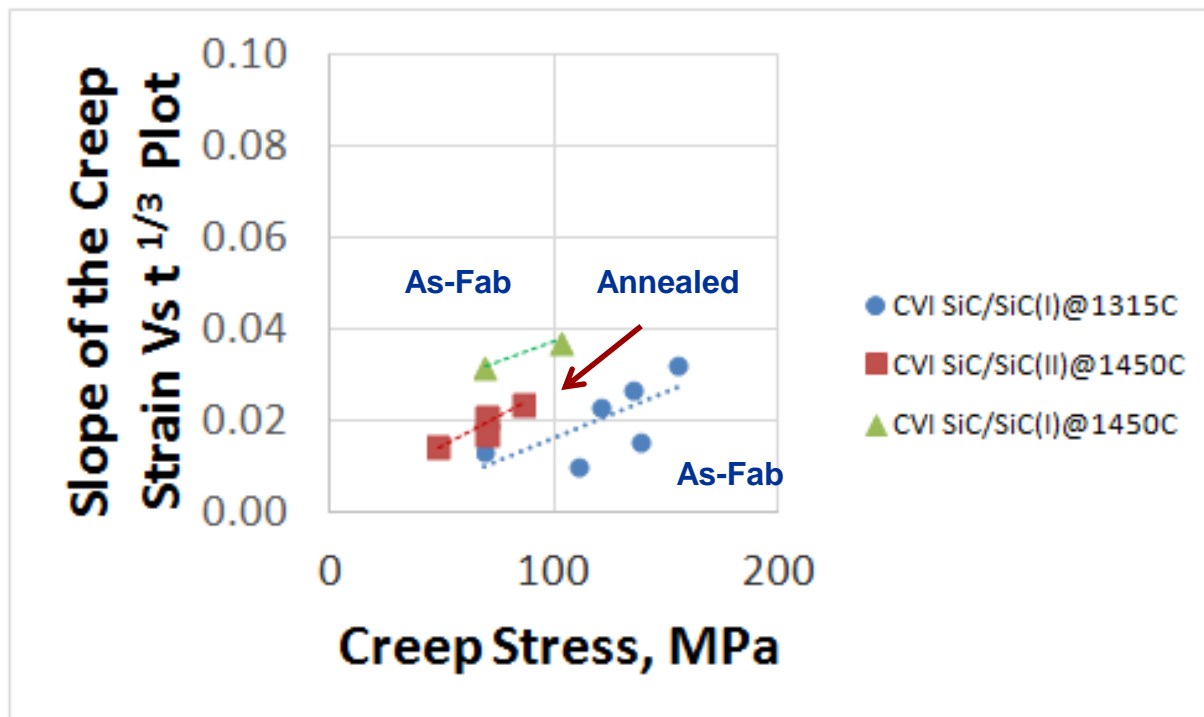
CVI SiC/SiC(II) at 1450°C



The creep strain of full CVI SiC/SiC composites varies linearly with time $(t^{1/3})$.



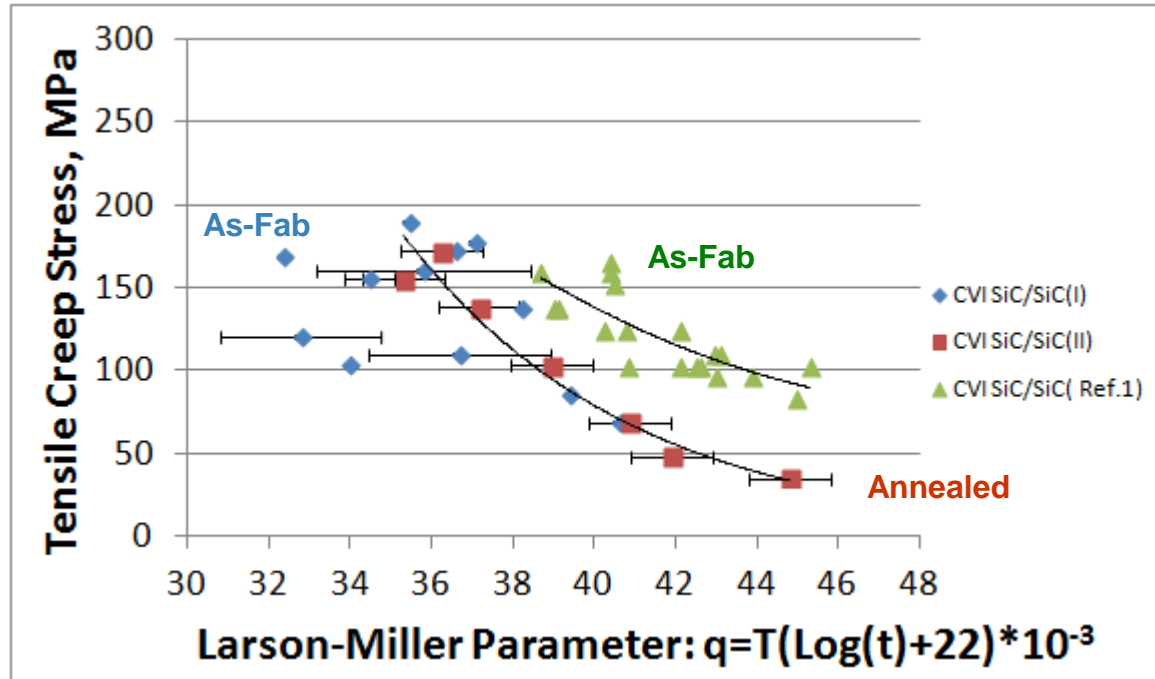
Influence of Stress/Temperature on Slope of the Creep Strain vs. $t^{1/3}$ Plot



The slope of the creep strain vs. time $(t^{1/3})$ is influenced by creep stress, temperature and composition of CVI SiC



Measured On-Axis Rupture Strength in Air for Full-CVI SiC/SiC CMCs with 2D 0/90-Balanced Fiber Architecture

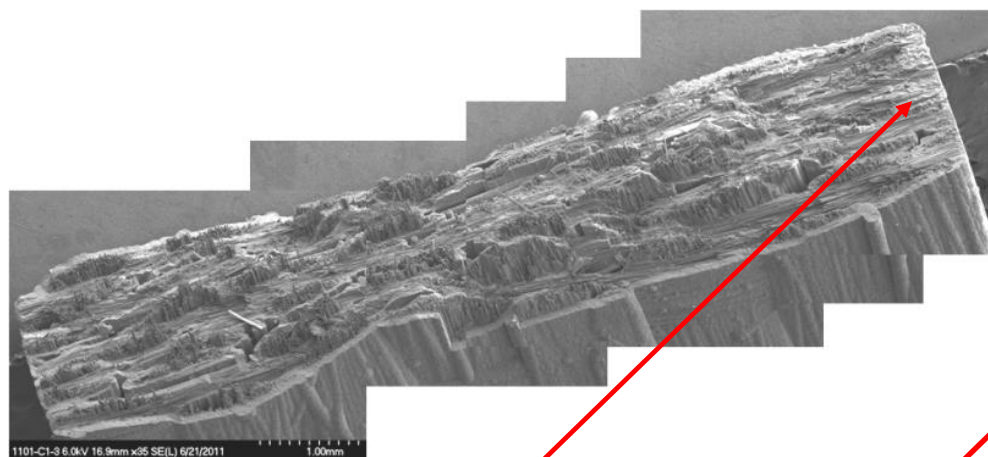


Significant variation in creep rupture life was observed at a fixed stress/temperature from lot to lot and within a lot depending on the CVI vendor and post processing heat treatment. T temperature in K and t time in hours

1. "SiC/SiC Composites for High Temperature Hypersonic Vehicle Structural Thermal Protection Systems"
R. Shinavski, W. Thibault, and S. Harris, NASA SBIR Phase II Final Report , 2014.

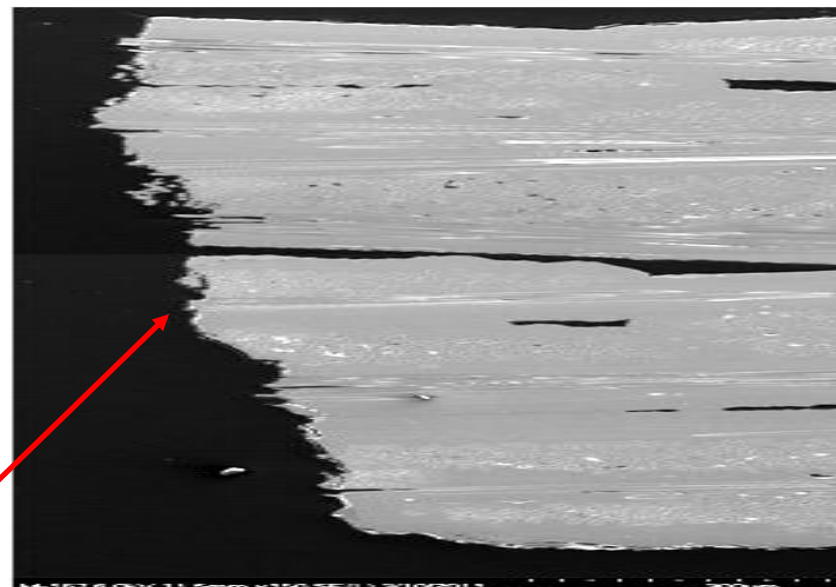
Fracture Analysis of Creep Ruptured Full CVI SiC/SiC Composites

**Tensile Fracture Surface
(1450°C/69MPa/58hrs/Air)**



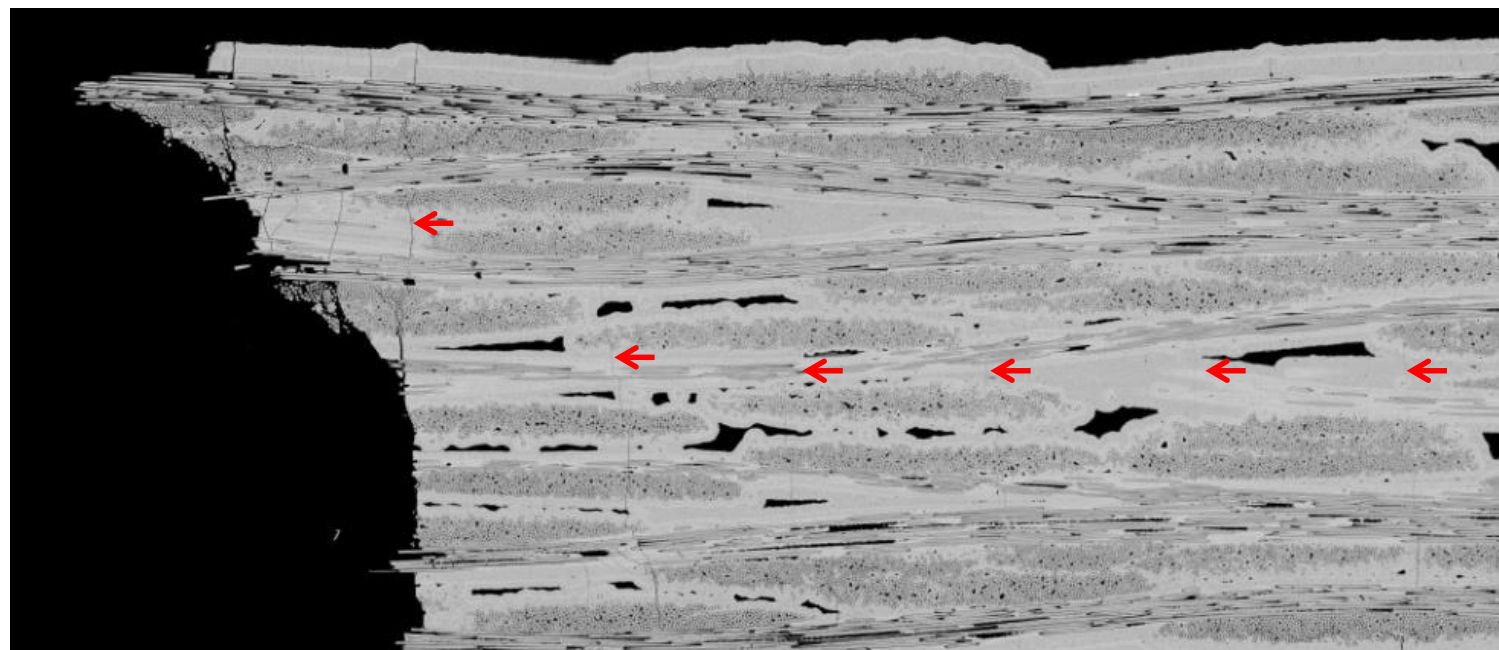
Possible crack initiation site

**Longitudinal Cross Section of the same
specimen**



All creep ruptured specimens show only one dominant crack.

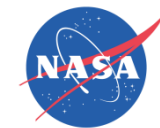
Longitudinal Cross Section of a CVI SiC/SiC Composite Specimen Tensile Creep Tested at 1450°C in Air at 69 MPa for 100hrs and then Fast Fracture Tensile Tested at the Same Temperature



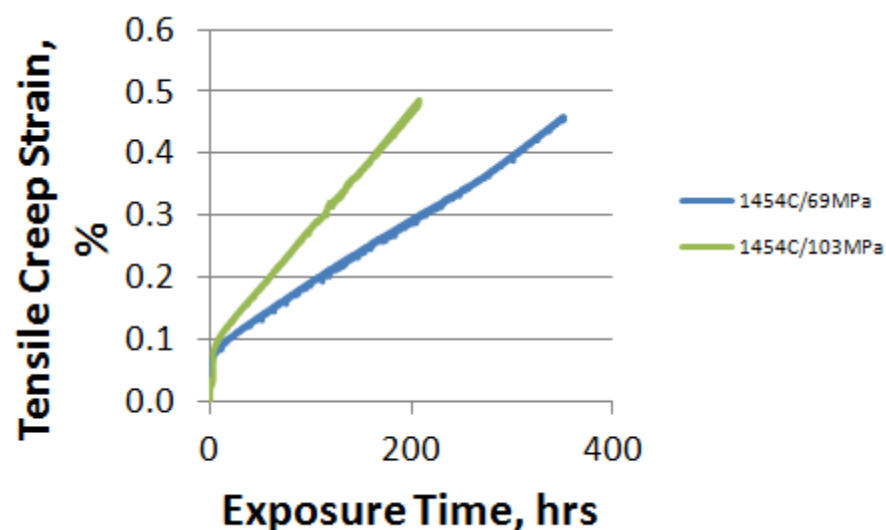
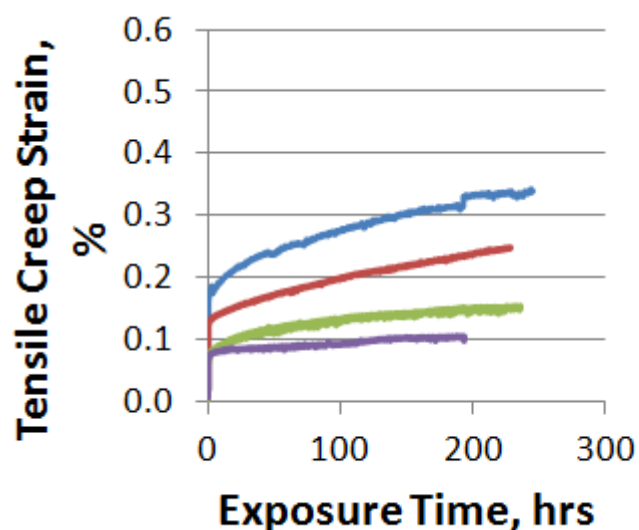
Tensile tested crept specimens show periodic through-the-thickness matrix cracks



Creep Properties of Full PIP SiC/SiC Composites



Influence of Stress on Creep Behavior of Full PIP SiC/SiC Composites at 1315°C and 1454°C in air

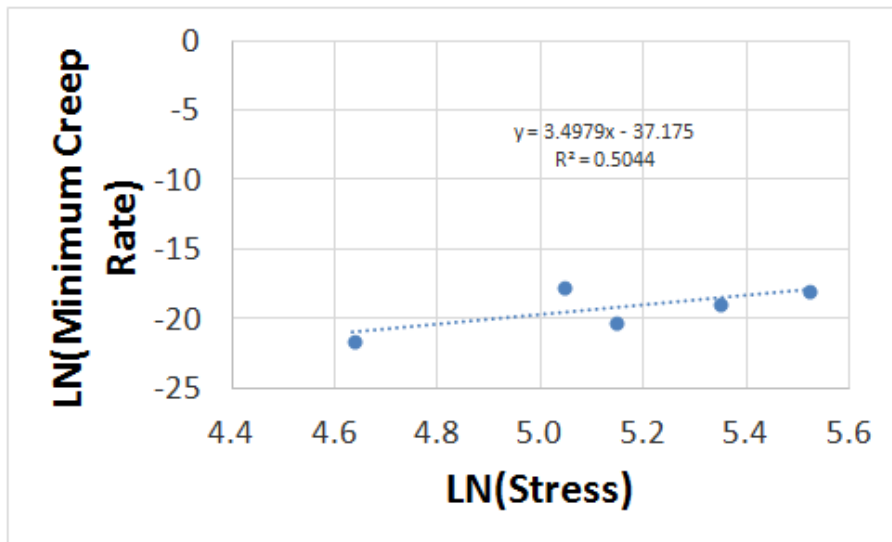


The creep strain in full PIP SiC/SiC composites varies linearly with time

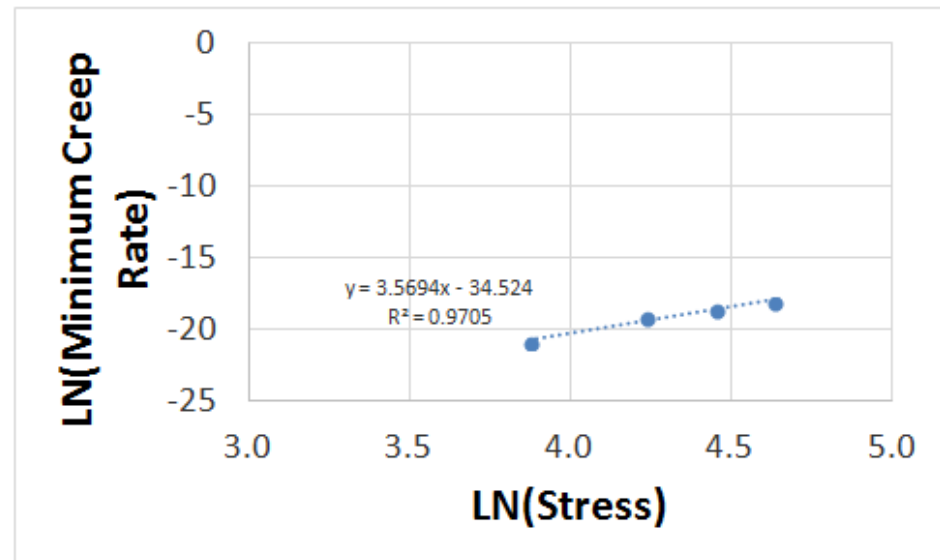


Stress Exponents of Creep for Full PIP SiC/SiC Composites at 1315°C and 1450°C

PIP SiC/SiC(I) at 1315°C



PIP SiC/SiC(II) at 1454°C

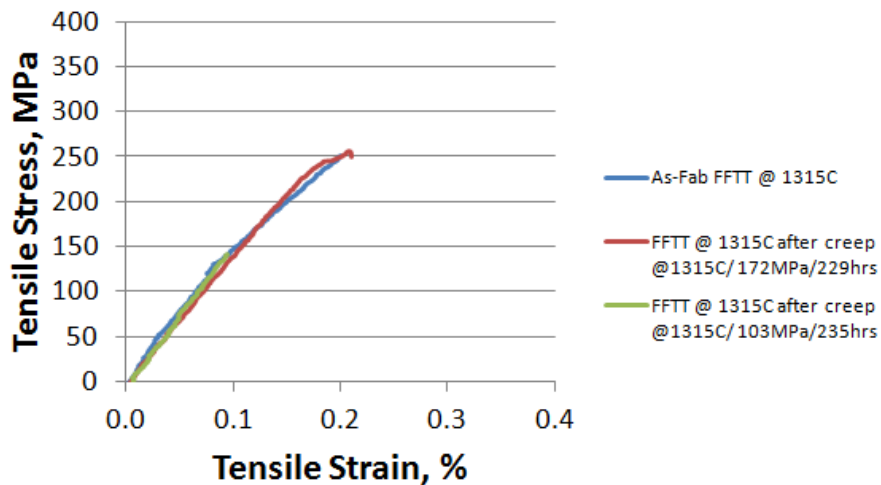


From 1315°C to 1450°C, the stress exponent of creep is ~3.5. For measuring stress exponents, creep data of specimens that survived >250hr exposure were chosen.

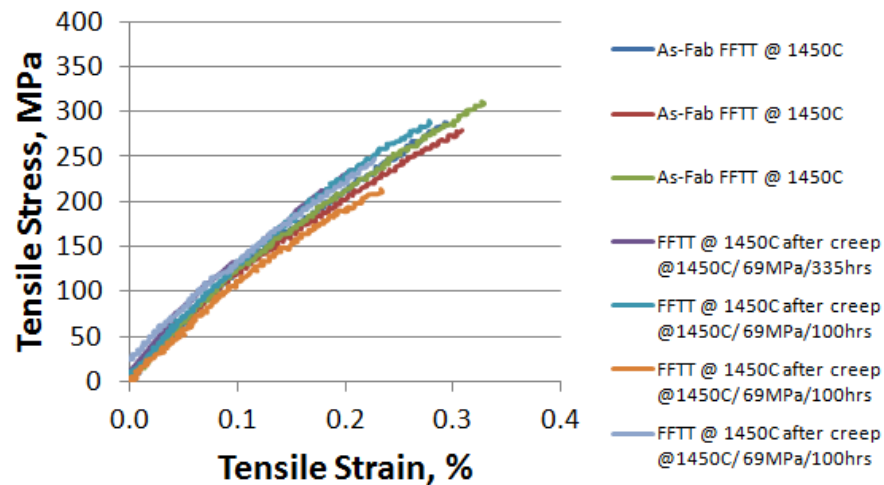


Influence of Accumulated Creep Strain on 1350°C and 1450°C Tensile Stress-Strain Behavior of Full PIP SiC/SiC Composites

Full PIP SiC/SiC(I)

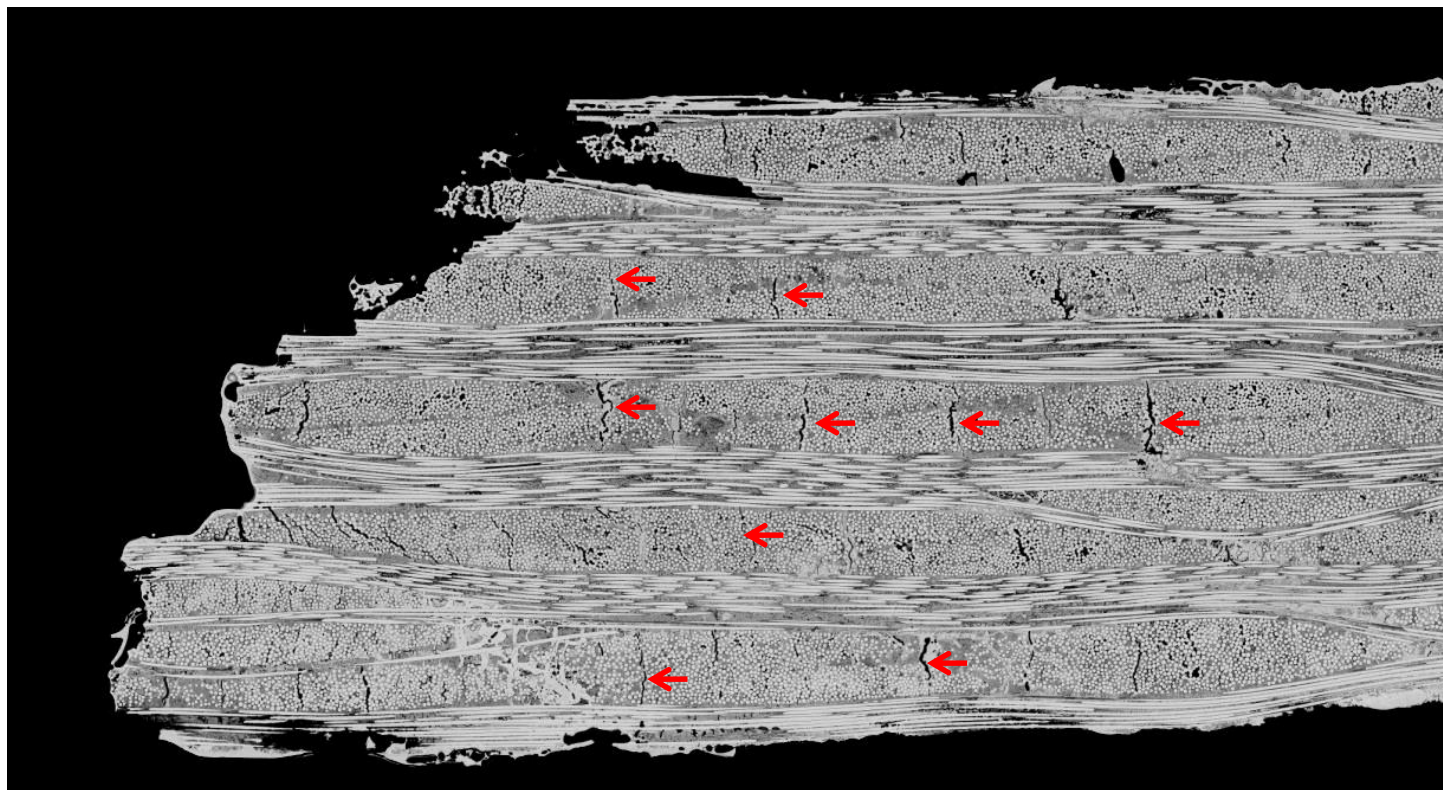


Full PIP SiC/SiC(II)



Crept specimens retain significant % of in-plane tensile properties of as-fabricated composites

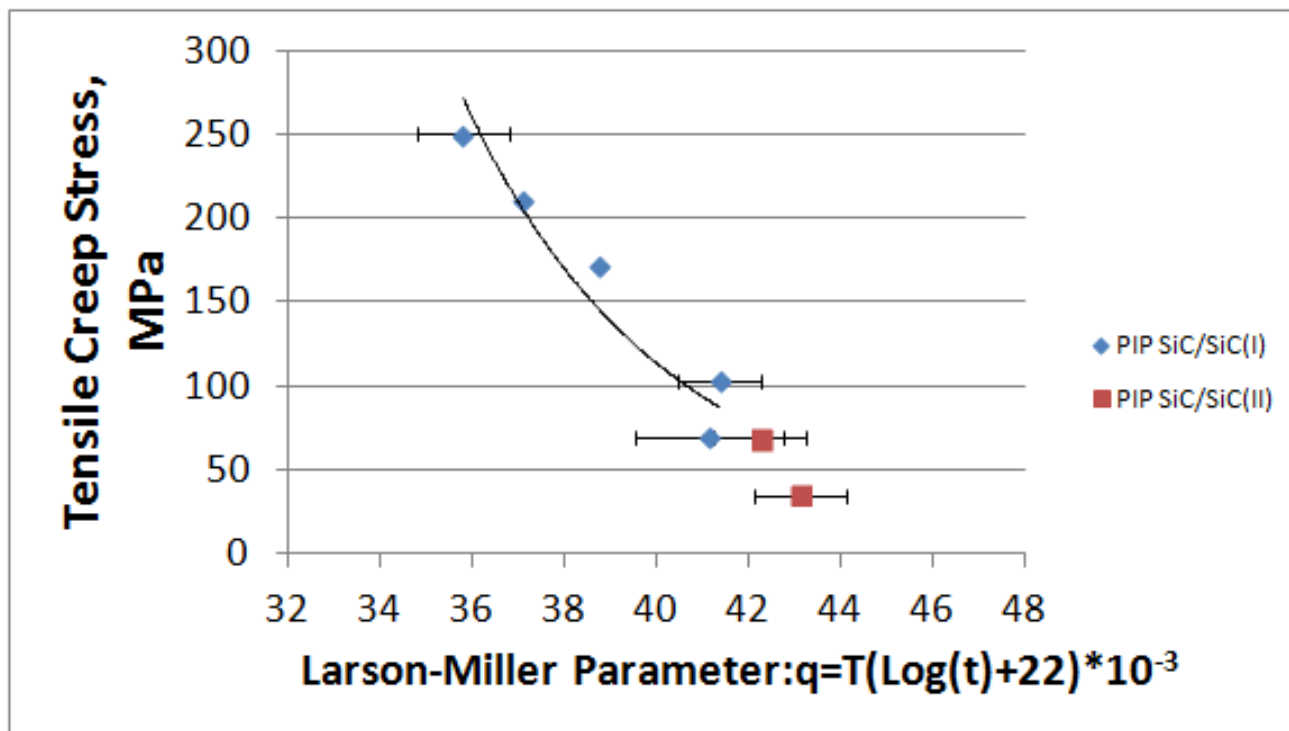
Cross Section a PIP SiC/SiC Composite Specimen Tensile Creep Rupture Tested at 1450°C in Air at 69 MPa for 115 hrs



Extensive tunnel cracks in the 90° tows (Red arrows). Significant oxidation of PIP matrix near the fracture surface as well as in the 90° tows.



Measured On-Axis Rupture Strength in Air for Full PIP SiC/SiC CMCs with 2D 0/90-Balanced Fiber Architecture



t = rupture time (hours) for constant stress creep-rupture test at temperature T (Kelvin)

Different batches of full PIP SiC/SiC composites exhibit similar creep rupture time when tested at the same stress/temperature.



Summary of Results

- **Creep deformation and rupture in full CVI SiC/SiC composites controlled by three factors: stress, temperature and creep mismatch ratio between the SiC fiber and CVI SiC matrix. At creep stresses above the matrix cracking stress, creep deformation is controlled by the fibers and below the matrix cracking stress by the CVI SiC matrix.**
- **Creep rate in this system continuously decreases with time of exposure and does not reach a steady state value.**
- **At a fixed creep stress below the matrix cracking stress, the time exponent for creep is 1/3 and the stress exponent for creep is between 1.5 to 2.5 from 1315°C to 1450°C.**
- **Creep rupture is caused by a single dominant crack which starts from specimen edge or corner or an internal flaw.**
- **Specimens that survived creep test retain significant % of as fabricated in-plane tensile properties when fast fracture tensile tested at room or at elevated temperature.**



Summary of Results-Continued

- In full PIP SiC/SiC composites, the matrix is micro-cracked and not load bearing. Therefore, the creep strain is predominantly controlled by the SiC fibers.
- The creep strain in this system follows linear dependency with time and shows a stress dependency of ~ 3.5
- At early stages of creep deformation, tunnel cracks are formed in the 90° tows causing 0° tows to bear all the load.
- Creep rupture is caused by a single dominant crack, mainly due to linkage of tunnel cracks and fracture of the weak 0° tows.



Conclusion

Based on the creep data generated in this study, it appears that creep behavior of 3D woven SiC/SiC composites with (CVI+PIP) hybrid matrix can be modeled using the creep parameters of 2D woven full CVI SiC/SiC composites. However, predicting creep rupture life of 2D or 3D woven SiC/SiC composites is difficult and is influenced by factors such as size and stress intensity of the dominant flaw, and crack growth kinetics.